

# PATENT SPECIFICATION

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## (54) OLEORESIN EMULSIONS

(71) We, BUSH BOAKE ALLEN LIMITED, a British Company, of Blackhorse Lane, Walthamstow, London E17 7QP, England, do hereby declare the invention, for which we pray that a Patent may be granted to us and performed to be particularly described in and by the following statement:—

This invention relates to oleoresin emulsions and processes for making them.

In our German OLS 2348004 we describe a process for preparing an emulsion of a flavour oleoresin which process comprises in step (a) forming a solution or colloidal solution of the oleoresin in a diluent, in step (b) mixing the solution or colloidal solution with agitation with a preformed mixture comprising an emulsion stabiliser, and at least one of an edible and essential oil, in step (c) adding an aqueous solution or dispersion of an emulsion stabiliser to form a final mixture, and in step (d) emulsifying the final mixture. Examples of the diluent are given as alcohols such as ethanol, isopropanol and glycerol and esters such as ethyl acetate.

We have now found a method of making emulsions and hence micro encapsulated products having an essential oil content which can be low and is variable at will, and simpler than the above method.

The present invention provides a process for preparing an emulsion of a flavour oleoresin which comprises in step (a) forming a solution of dispersion of an oleoresin having a non volatile component, which is pepper, cinnamon or turmeric oleoresin, in 1—30 parts of an acid of formula RCOOH, where R is hydrogen, methyl or ethyl, and 0—85 parts of a flavour essential oil, the parts of acid and essential oil (if present) being by weight per 100 parts of non volatile component of the oleoresin, and in step (b) mixing and emulsifying this solution or dispersion with an aqueous solution or colloidal solution of an emulsion stabilizer.

According to a further feature of the invention the emulsion, when containing an encapsulant as emulsion stabilizer or added

separately, is dried to give a micro-encapsulated product for flavouring food, often in association with other flavouring agents.

The flavour oleoresins are compounds obtained from the appropriate plant material and extractable therefrom by solvent extraction and subsequent removal of the solvent and optionally volatile flavour components. The oleoresins contain the non volatile components of the extract and an amount of volatile components which can vary from zero to about 30%, usually 10—25%, based on the total weight depending on the nature of the oleoresin, the method of extraction and subsequent treatment; e.g. cinnamon oleoresin may contain virtually no essential oil.

The flavour essential oils are volatile compounds obtained from an appropriate plant material and extractable therefrom by many methods including cold pressing, distillation, steam distillation and evaporation of the solvent, followed if desired by separation of the volatile components.

Normally the essential oil is derived from the same plant as the oleoresin; thus for pepper oleoresin pepper oil or at least one fraction thereof is used and for turmeric oleoresin, turmeric oil or at least one fraction thereof is suitable. Mixtures of essential oils or fractions thereof can of course be used. Usually a pepper oleoresin containing about 20% essential oil or a turmeric oleoresin containing about 25% essential oil is used and further essential oil is added if necessary to bring the essential oil content in the emulsion and micro encapsulated product to the desired level. Thus for example solutions or dispersions of 100 parts of such oleoresins containing 16—25% e.g. 16—20% of inherent essential oil (based on the total weight of oleoresin) and correspondingly 84—75% e.g. 84—80% of non volatile component with 2—12 e.g. 2—10, preferably 3—6 or 5—12 parts of the acid and 0—40 e.g. 3—40, preferably 7—35 parts, or 0—10 parts of added essential oil can be

produced in step (a) and formed according to the process of the invention into emulsions and micro encapsulated products having highly desirable flavour characteristics. For emulsions with a medium or low essential oil content, the solutions or dispersions of 100 parts of such oleoresins in step (a) most preferably contain 5—12 parts of the acid and 0—10 parts of added essential oil. For emulsions with a medium or high essential oil content the solutions or dispersions of 100 parts of oleoresin most preferably contain 3—6 parts of acid and 7—35 parts of added essential oil.

However, the process of the invention is not restricted to the use of such oleoresins and may also be applied to pepper or turmeric oleoresins containing the 10—15% essential oil also commonly obtained and also pepper, cinnamon or turmeric oleoresin containing substantially no essential oil. Both the essential oil and the acid act as solvents for the non volatile component of the oleoresin and hence, if the oleoresin contains no essential oil volatile component, then more acid per unit weight of oleoresin is needed than is the case if the oleoresin contains essential oil. We have found that solutions or dispersions of oleoresin with 2—15, e.g. 2—10 and preferably 4—7.5 or e.g. 6—15 parts of acid and 15—70 e.g. 20—70 and preferably 30—65, especially 35—65 or e.g. 15—40 and preferably 15—30 parts of essential oil per 100 parts of non volatile component of the oleoresin are highly satisfactory for forming emulsions and micro encapsulated products with the required flavour characteristics. For emulsions without a high essential oil content, solutions or dispersions of 100 parts of non volatile component of oleoresin and 6—15 parts acid and 15—40, especially 15—30 parts total essential oil are preferred. For emulsions without a low essential oil content solutions or dispersions of 100 parts of non volatile component of oleoresin and 4—7.5 parts acid and 30—65, especially 35—65 parts of total essential oil are preferred. If the emulsion is to contain no essential oil, then in step (a) the solution or dispersion usually contains at least 6 parts of acid per 100 parts of the oleoresin, which has no volatile essential oil. A solution is preferably formed in step (a), especially when the oleoresin is pepper oleoresin.

The acid mixed with the oleoresin is formic, acetic or propionic acid but is preferably acetic acid, which is usually added as glacial acetic acid, though acetic acid of at least 80% content may be used.

Normally heating of the oleoresin and solvent is needed to prepare the solution or dispersion in step (a) of the process, e.g. heating to 80—110°C such as about 90°C. Usually the oleoresin and acid are heated

first until a solution or dispersion is achieved and then the extra essential oil (if any) is added. The solution or dispersion produced usually contains 10%—30% preferably 10%—25% and especially 10—20% by weight of acid and added essential oil, based on the total weight of the oleoresin (i.e. the non volatile content and the inherent essential oil, when the latter is 10—25% of the non volatile content).

In step (b) of the process, the solution from step (a) is mixed and emulsified with an aqueous solution or colloidal solution of an emulsion stabilizer with continuous vigorous agitation.

The emulsion stabilizer is usually one which can act as an encapsulant for the oleoresin and essential oil (if present) in the production of microencapsulated products, e.g. by spraydrying, but if the emulsion is to be used as such for flavouring the stabilizer need not be an encapsulant. Some of the stabilizers also act as the emulsifying agent required to be present in the aqueous solution or colloidal solution; examples of such stabilizers are natural or modified gums such as gum acacia, pectins, alginates, proteinaceous materials such as gelatin or casein, both natural and modified, and carrageenins. The stabilizer encapsulants, which do not act as emulsifying agents and therefore require added emulsifying agents include starch based compounds such as natural or modified starch e.g. dextrinized starch or phosphorylated starch, dextrin, maltodextrin and wet milled flour (usually available in slurry form).

The emulsifying agent, which must be added to the aqueous solution or colloidal solution of stabilizer if the latter does not have emulsifying properties, is usually present in an amount of up to 15% e.g. 5—15% by weight based on the weight of the stabilizer. Examples of such agents are lecithins, glycerol fatty acid esters, milk whey, polysorbates e.g. fatty acid partial esters of sorbitol anhydride or sorbitan (such as those sold under the Trade Mark "Span"), polyoxyethylene derivatives of fatty acid partial esters of sorbitol anhydride (such as those sold under the Trade Mark "Tween"), mono- and di-ethyl esters of di- and tri-carboxylic acids of 2 to 6 carbon atoms and any of the encapsulants which are also emulsifying agents. Milk whey solids is the preferred emulsifying agent. Also the presence of 2—20% e.g. 9—15% by weight (based on the weight of stabilizer) of pectin as well as the stabilizer and other emulsifying agent has been found to increase the stability of the oleoresin emulsion obtained.

The encapsulant and emulsifying agent must comply with any relevant food regulations.

If the emulsion is to be dried to give a microencapsulated product it is necessary that it contains an encapsulant, which is preferably one of the stabilizers, e.g. maltodextrin, but may be added separately.

The aqueous solution or colloidal solution in step (b) is usually warmed before mixing with the solution or dispersion of oleoresin, temperatures of 50–70°C especially 50–60°C being preferred. Usually the oleoresin solution/dispersion and aqueous solution or colloidal solution are mixed in a weight ratio of 1:1.5 to 1:10 e.g. 1:4 to 1:8. The aqueous solution or colloidal solution of emulsion stabilizer preferably contains 0.5–15 usually 2–6 and especially 3–5 parts water per part of stabilizer and 1–12, especially 1.5–4 parts of stabilizer per part of non volatile component of oleoresin. The conditions of mixing are chosen so that there is no immediate deposition of solid material on mixing the oleoresin solution and aqueous phase, which tends to occur when a concentrated oleoresin solution is added hot to a small quantity of cold aqueous phase.

The emulsification is carried out by conventional techniques such as mechanical emulsification, for example in a turbine mixer, for instance, a Silverson Mixer, although other mechanical means for preparing the emulsion may be employed if desired or ultrasonic emulsification may be used. Usually the mixing and emulsification are performed in the same apparatus.

Examples of the emulsions prepared in step (c) are those comprising 8–40%, preferably 20–40% e.g. 20–30% of non-volatile component of oleoresin, 0–20% usually 3–20 e.g. 3–9 or e.g. 5–20 preferably 9–17% of total essential oil, 20–75%, e.g. 40–68% stabilizer, 0.5–5% preferably 0.8–4% e.g. 0.8–2.5 or 1.5–4% of the acid and up to 13% e.g. 2–10% of emulsifying agent the percentages being by weight based on the weight of the total non aqueous components and the higher percentages of acid usually being found with the lower percentages of total essential oil and vice versa. The presence of the acid does not generally detract from the overall flavour of the emulsion (or dried product produced therefrom) in view of the dominant effect of the oleoresin and essential oil (if present).

Emulsion prepared according to the invention are stable e.g. for at least a week and often for a month or more and are particularly suitable for use directly as food flavouring or in the production of micro encapsulated flavouring materials by spray drying or other drying means. When the emulsion contains an encapsulant, e.g. maltodextrin, it may be spray dried by known methods e.g. by using a spray drier — atomizer or spinning disc under conditions and with a flow rate sufficient to

provide an inlet temperature of 175–210°C and an outlet temperature of 90–110°C; inlet and outlet temperatures may be at 200°C and about 100°C respectively are usual. In the act of spray drying substantial amounts of the acid are evaporated leaving a dried product. The microencapsulated particles usually have a lower degree of cohesion than those made according to the method described in OLS 2348004 when propylene glycol or glycerol is the diluent. The flavours can be used in e.g. sauces, soups, processed meat products, and vinegar, with the emulsions usually being used for the flavouring of wet foods.

The invention is illustrated in the following examples in which parts are by weight.

#### EXAMPLE 1

A mixture of 30 parts pepper oleoresin (containing 24 parts non volatile component and 6 parts pepper essential oil), 1 part glacial acetic acid and 9 parts pepper oil was refluxed at about 80–90°C until solution occurred. The solution obtained was added hot to a solution of 50 parts of maltodextrin, 5 parts of whey powder, 5 parts of pectin and 200 parts of water at 50°C with continuous stirring and the mass obtained emulsified in a Silverson Mixer. The emulsion obtained was stable for at least a week, and could be spray dried to give microencapsulated pepper flavour particles with a low degree of cohesion.

#### EXAMPLE 2

The process of Example 1 was repeated but with 35 parts pepper Oleoresin (containing 28 parts non volatile component and 7 parts pepper essential oil), 2 parts of acetic acid and 3 parts of pepper oil. An emulsion stable for at least a week was obtained and from it by spray drying micro encapsulated pepper flavour particles with a low degree of cohesion.

#### EXAMPLE 3

The process of Example 1 was repeated but with 35 parts of pepper oleoresin (containing 29 parts of non volatile components and 6 parts of pepper essential oil), 2 parts of acetic acid and 1.5 parts of added pepper essential oil, to replace the amounts used in Example 1. A stable emulsion was prepared which could be spray dried as before.

#### EXAMPLE 4

The process of Example 1 was repeated but with 35 parts of pepper oleoresin (containing 29 parts of non volatile components and 6 parts of pepper essential oil), 3.5 parts of acetic acid and no added pepper essential oil, to replace the amounts used in

Example 1. A stable emulsion was prepared which could be spray dried as before.

# WHAT WE CLAIM IS:—

1. A process for preparing an emulsion of a flavour oleoresin, which comprises in step (a) forming a solution or dispersion of an oleoresin, having a non volatile component, which oleoresin is pepper, cinnamon or turmeric oleoresin, in 1 to 30 parts of an acid of formula  $\text{RCOOH}$ , where R is hydrogen, methyl or ethyl, and 0—85 parts of a flavour essential oil, the parts of acid and essential oil (if present) being by weight per 100 parts of non volatile component of the oleoresin, and in step (b) mixing and emulsifying this solution or dispersion with an aqueous solution or colloidal solution of an emulsion stabilizer.

2. A process according to Claim 1 wherein the solution or dispersion in step (a) contains 2—15 parts of acid and 15—70 parts of essential oil per 100 parts of non volatile component of the oleoresin.

3. A process according to Claim 2 wherein the solution or dispersion contains 6—15 parts of acid and 15—30 parts of essential oil per 100 parts of non volatile component of the oleoresin.

4. A process according to Claim 2 wherein the solution or dispersion contains 4—7.5 parts of acid and 30—65 parts of essential oil per 100 parts of non volatile component of the oleoresin.

5. A process according to any one of Claims 1—4 wherein the oleoresin is pepper oleoresin and a solution is formed in step (a).

6. A process according to any one of Claims 1—5 wherein the oleoresin contains 10—25% of essential oil and 90—75% of non volatile component.

7. A process according to Claim 6 wherein the solution or dispersion in step (a) contains 10—30% of the acid and added essential oil (if any) as a percentage of the total weight of added oleoresin.

8. A process according to Claim 7 wherein the solution or dispersion in step (a) contains 10—20% of acid and added essential oil (if any) as a percentage of the total weight of added oleoresin.

9. A process according to Claim 6 which comprises in step (a) forming a solution or dispersion of pepper oleoresin containing 16—10% essential oil and 84—80% non volatile component in 2—10 parts of acid and 0—40 parts of extra essential oil per 100 parts of the total oleoresin.

10. A process according to any one of the preceding claims wherein the acid is acetic acid.

11. A process according to any one of the preceding claims which comprises in step (a) forming the solution or dispersion by

heating the oleoresin, acid and essential oil (if any) at 80—110°C.

12. A process according to any one of the preceding claims which comprises in step (b) mixing the solution or dispersion from step (a) with the aqueous solution or colloidal solution at 50—70°C.

13. A process according to any one of the preceding claims wherein the aqueous solution or colloidal solution is mixed with the solution or dispersion from step (a) in a weight ratio of 1.5:1 to 10:1, the aqueous solution or colloidal solution contains 0.5 to 15 parts water per part of stabilizer and 1 to 12 parts of stabilizer per part of non volatile component of the oleoresin.

14. A process according to any one of the preceding claims wherein the stabilizer is maltodextrin or gum acacia, and when the stabilizer is maltodextrin, the aqueous solution or colloidal solution contains 5 to 15% by weight (based on the weight of stabilizer) of an emulsifying agent.

15. A process according to any one of the preceding claims wherein the aqueous solution or colloidal solution contains 2—20% by weight (based on the weight of stabilizer) of pectin.

16. A process according to any one of the preceding claims wherein an emulsion comprising 8—40% of non volatile component of oleoresin, 5—20% in total of essential oil, 20—75% stabilizer, 0.5—5% of the acid and up to 13% emulsifying agent is formed.

17. A process according to Claim 1 substantially as described in Example 1 or 2.

18. A process according to Claim 1 substantially as described in Example 3 or 4.

19. An emulsion obtained by a process according to any one of the preceding claims.

20. An emulsion obtained by a process according to any one of Claims 1, 2, 4 and 10—14 when appendant thereto, and 17.

21. An aqueous flavour oleoresin emulsion which comprises 8—40% of non volatile component of pepper, cinnamon or turmeric oleoresin, 0—20% in total of essential oil, 20—75% of stabilizer, 0.5—5% of an acid of formula  $\text{RCOOH}$ , where R is hydrogen, methyl or ethyl and 0 to 13% of emulsifying agent, the percentages being by weight based on the total weight of non aqueous components of the emulsion.

22. An emulsion according to Claim 21 wherein the oleoresin and essential oil are derived from pepper.

23. An emulsion according to Claim 21 or 22 which comprises 20—30% of non volatile component of oleoresin, 5—20% of total essential oil, 40—68% of stabilizer and 0.8—4% of acid.

24. An emulsion according to Claim 21, 22 and 23 wherein the stabilizer is starch based,

- and the emulsion also comprises 5 to 15% by weight (based on the weight of stabilizer) of an emulsifying agent.
- 5 25. An emulsion according to any one of Claims 21—24 which also comprises 2—20% of pectin (based on the weight of stabilizer.
- 10 26. An emulsion according to Claim 21 substantially as described in Example 1 or 2.
27. An emulsion according to Claim 21 substantially as described in Example 3 or 4.
28. A flavouring composition which comprises an emulsion according to any one of Claims 19—27 and a flavouring agent.
- 15 29. A microencapsulated flavouring product obtained by drying an emulsion according to any one of Claims 19—27, or a flavouring composition according to Claim 28, each of which contains an encapsulant.
30. A product according to Claim 29 obtained from an emulsion according Claim 20.
31. A food flavoured with an emulsion according to any one of Claims 19—27 or a flavouring composition according to Claim 28.
32. A food flavoured with a micro-encapsulated product according to Claim 29 or 30.
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